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MEMORANDUM

DATE:

18 January 1999

TO

David Bennett, WAM, U.S. EPA, Region X

FROM:

Michelle Turner, Chemist, WESTON, Seattle

Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT:

Validation of Organotin Data

Laboratory Batch: K9805863

Site: Duwamish River

WORK ASSIGNMENT NO

46-35-0JZZ

WORK ORDER NO.:

4000-019-038-5200-00

DOC CONTROL NO: 4000-019-038-AAAK

cc:

Bruce Woods, RAP-WAM, U.S. EPA, Region X

Dena Hughes, Site Manager, WESTON, Seattle (memo only) Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of four sediment samples, laboratory batch K9805863, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98354077

98364000

98364014

98364018

Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the National Functional Guidelines for Organic Data Review (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods

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1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples were extracted 9 or 13 days after sample collection, exceeding the 7 day holding time criteria. Prior to extraction, samples were stored frozen, thus extending the holding time. Samples were extracted within the 12 month holding time recommended by PSEP for frozen samples. However, samples were analyzed 8 days after extraction and exceeded analysis holding times as follows:

Sample ID	Date Collected	Date Extracted	Date Analyzed	No of Days
98354077	8/27/98	9/9/98	9/17/98	13 days (extraction) 8 days (analysis)
98364000	8/31/98	9/9/98	9/17/98	9 days (extraction) 8 days (analysis)
98364014	8/31/98	9/9/98	9/17/98	9 days (extraction) 8 days (analysis)
98364018	8/31/98	9/9/98	9/17/98	9 days (extraction) 8 days (analysis)

Results and quantitation limits for the above samples have been qualified as estimated (UJ/J).

2 Detection Limits

Detection limits met project required quantitation limits with the following exceptions:

Sample	Compound	QL Goal (µg/Kg)	Reported QL (μg/Kg)
98354077	n-Butyltın	10	35

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference.

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3. Initial Calibration

A seven-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. Target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent (75-125 percent recovery) with the following exceptions:

Sample	Compound	% Recovery	Associated Samples
CCV2	Dibutyltin	131	none

As no samples were directly associated with samples from this SDG, no qualifiers were assigned based on continuing calibration results

Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this SDG.

6 Surrogate Compound Recovery

Surrogate recovery goals for Tripropyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment. Based on conversations with the laboratory an additional surrogate, Tripentyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below.

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Surrogate Compound	Sediment Limits
Tripropyltin	20 - 195%
Tripentyltin	20 - 172%

Surrogate compound percent recoveries exceeded the QC limits for the following samples:

Sample	Surrogate	% Recovery
98354077	Tripropyltin	59

Samples results and detection limits were qualified as estimated (UJ/J) when both surrogate recoveries were outside the QC limits.

7. Laboratory Control Sample (LCS)

LCS recovery goals for Butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment Based on conversations with the laboratory, historical control chart limits of 20 to 164 percent for sediment were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the following exceptions:

LCS	Analyte	% Recovery	QC Limit	Associated Samples
K980909-LCS	n-Butyltın	34	60-130 (P) 20-164 (L)	98354077 98364000 98364001 98364014
K980909-DLCS	n-Butyltın	24	60-130 (P) 20-164 (L)	98354077 98364000 98364001 98364014

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Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits.

8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

A MS/MSD analysis was not performed for this SDG. Instead, a LCS/DLCS was analyzed for quality control.

9. Field Duplicate Analysis

No field duplicates were included with this SDG.

10. Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete. No problems were noted in the case narrative

11. Laboratory Contact

No laboratory contact was required.

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

Data Qualifiers

- U The compound was analyzed for, but was not detected
- UJ The compound was analyzed for, but was not detected The associated quantitation limit is an estimate because quality control criteria were not met
- The analyte was positively identified, but the associated numerical value is an
 estimated quantity because quality control criteria were not met or because
 concentrations reported are less than the quantitation limit or lowest calibration
 standard.

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- R Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- N Presumptive evidence of presence of material (tentative identification).
- I Elevated reporting limit due to matrix interference.

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Analytical Report

Client:

Roy F Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805863

Date Collected: 8/27/98

Date Received: 8/28/98

Butyltins

Sample Name

98354077

Lab Code

K9805863-003

Test Notes

D

Units ug/Kg (ppb)

Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltın	Method	Butyltins	5	5	9/9/98	9/17/98	ND WJ	
Trı-n-butyltın	Method	Butyltins	5	5	9/9/98	9/17/98	15 T	
Dı-n-butyltın	Method	Butyltins	5	5	9/9/98	9/17/98	ND WJ	
n-Butyltin	Method	Butyltuns	35	5	9/9/98	9/17/98	TU DN	B

В

The MRL is elevated because of matrix interferences

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Date 10/14/99

Analytical Report

Client:

Roy F Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805863

Date Collected: 8/31/98

Date Received: 9/1/98

Butyltins

Sample Name

98364000

Lab Code

K9805863-019

Test Notes

D

Units ug/Kg (ppb)

Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	ND UJ	
Trı-n-butyltın	Method	Butyltins	5	5	9/9/98	9/17/98	144 J	
Dı-n-butyltın	Method	Butyltins	5	5	9/9/98	9/17/98	32	
n-Butyltin	Method	Butyltuns	5	5	9/9/98	9/17/98	22 🗸	

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By

LS22/020597p

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Analytical Report

Client:

Roy F. Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805863

Date Collected: 8/31/98

Date Received: 9/1/98

Butyltins

Sample Name

98364014

Lab Code

K9805863-009

Test Notes.

D

Units ug/Kg (ppb)

Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltıns	5	5	9/9/98	9/17/98	ND UJ	
Trı-n-butyitın	Method	Butyltins	5	5	9/9/98	9/17/98	115 J	
Dı-n-butyltın	Method	Butyltıns	5	5	9/9/98	9/17/98	29	
n-Butyltın	Method	Butyltıns	5	5	9/9/98	9/17/98	19 🗸	

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The MRL is elevated because of matrix interferences and because the sample required diluting

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Analytical Report

Client:

Roy F Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805863

Date Collected: 8/31/98 Date Received: 9/1/98

Butyltins

Sample Name Lab Code

98364018

K9805863-013

Units ug/Kg (ppb)

Test Notes

D

Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	9/9/98	9/17/98	ND WJ	
Trı-n-butyltın	Method	Butyltıns	5	5	9/9/98	9/17/98	21 J	
Dı-n-butyltın	Method	Butyltins	5	5	9/9/98	9/17/98	7	
n-Butyltın	Method	Butyltins	5	5	9/9/98	9/17/98	8 🗸	

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By

1822/020597p

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Date 10/14/98